Solventless synthesis of iridium(0) nanoparticles

By
R. Redón[1] , F. Ramírez-Crescencio[1] and A. L. Fernández-Osorio[2]

Journal of Nanoparticle Research
Volume 13, Number 11 (2011), 5959-5965
DOI: 10.1007/s11051-011-0413-0
Published online: **15 May 2011**

Special Issue: Nanostructured Materials 2010

(1) CCADET, Universidad Nacional Autónoma de México, Cd. Universitaria, A.P. 70-186, C.P. 04510, México D. F., México.
(2) FES, Cuautitlán, Universidad Nacional Autónoma de México, Edo. de México, México.

Shridevi S Bhat
CY12D031

INTRODUCTION

- In the literature, there are several approaches for obtaining Ir(0) nanoparticles.

- Fonseca et al. *2003* synthesised Ir(0) nanoparticle by the reduction of metallic compounds by molecular hydrogen dissolved in Imidazolium ionic liquid.

- The use of metallic carbonyls and ionic liquids immersed in an argon atmosphere (Krämer et al. *2008*; Redel et al. *2009*; Redel et al. *2010*; Vollmer et al. *2010*).

- There are other approaches aside from the ionic liquids approach, i.e., using inverse micelles with SiO_2 as support (Miyao et al. *2005*).

- There is also the approach of including dendrimers and alumina as supports to protect the obtained nanoparticles (López-De Jesús et al. *2008*).

INTRODUCTION

- In the case of the sol–gel method, there has also been some research conducted with iridium under inert atmospheres (Birss et al. *1999*).

- There are methods that avoid the use of solvents in the process to obtain Ir(0) nanoparticles, called a mechanochemical process (Yang et al. *2006*; Delogu et al. *2003*; Šepelák et al. *2006*; Ennas et al. *2004*; Ivanov and Suryanarayana *2000*).

In this paper:

- The results of a solvent-free synthesis of iridium(0) nanoparticles is presented.

- The effect of the variation in reducing agent concentration and the annealing temperature used after the reaction was studied.

SYNTHESIS

$IrCl_3 + NaBH_4$

Mixed using agate mortar
15 min

Black powder

Heated in N_2 atmosphere
200 ^oC, 2 h.

Ir(0) NPs

Schematic of the procedure used.

RESULTS AND DISCUSSION

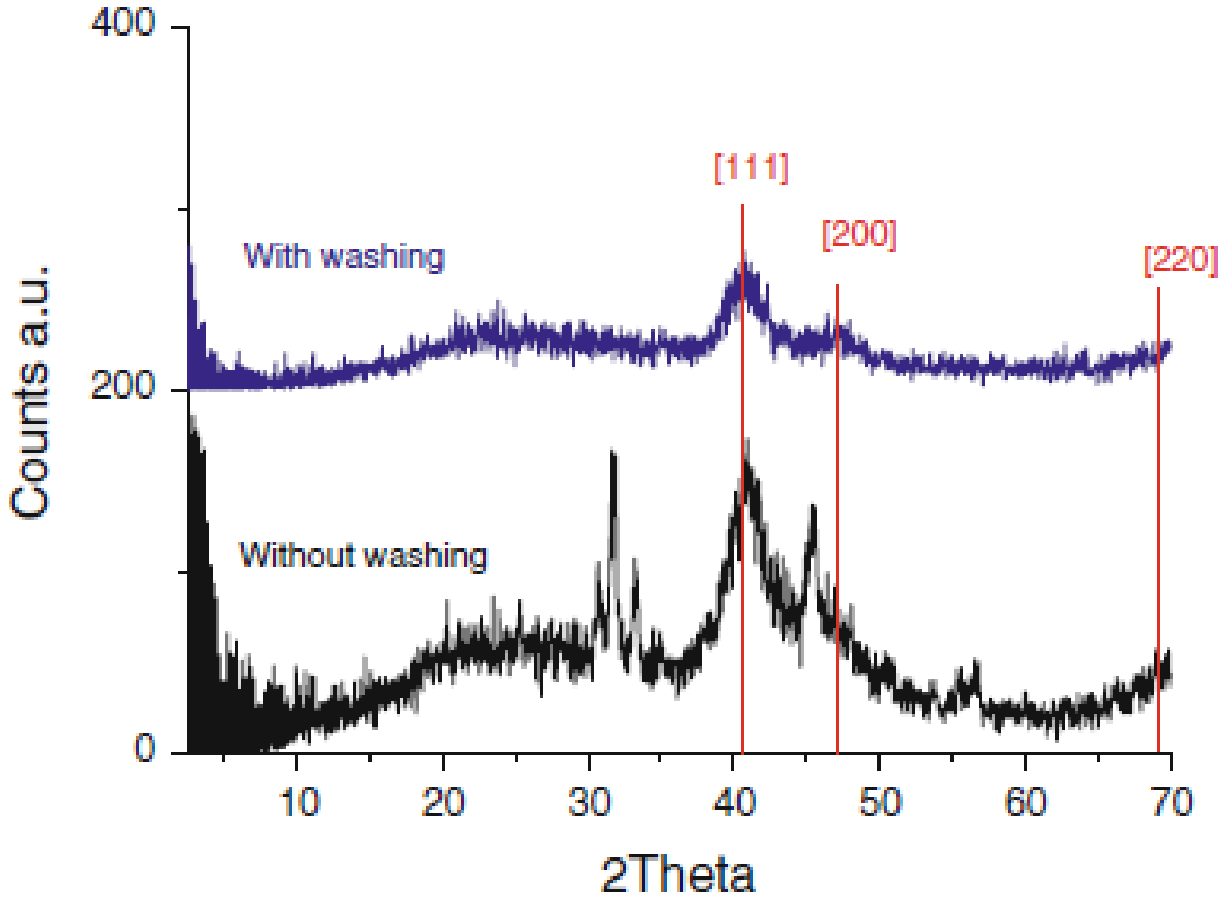

Fig. 1 X-ray powder diffractograms of Ir(0) NP samples before (black spectrum) and after washing (blue spectrum). The red bars correspond to bulk Ir metal [planes (111), (200), and (220) card JCPDS-ICDD 6-598].

RESULTS AND DISCUSSION

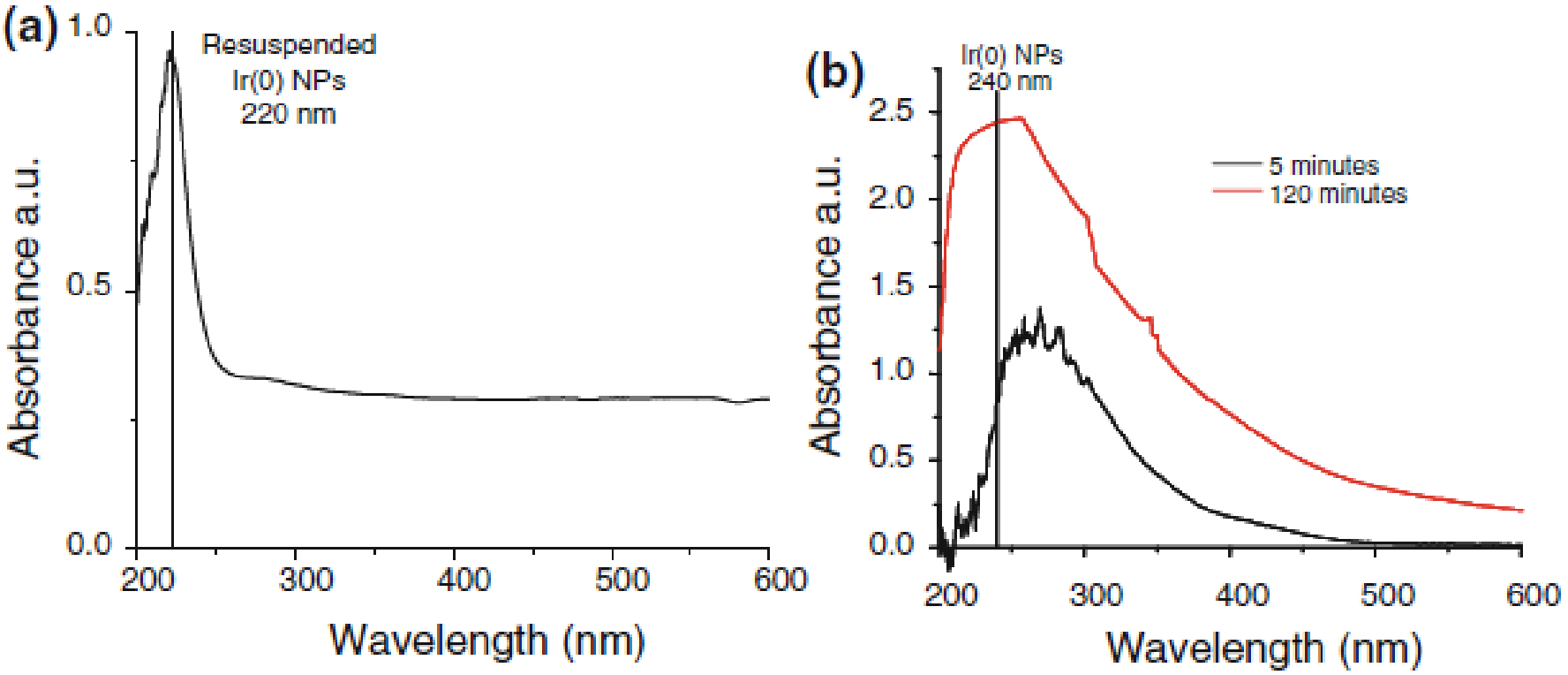

Fig. 2 a Absorption spectrum of Ir(0) nanoparticles suspended in ethylene glycol (220-nm absorption assigned to Ir(0) NPs). b Five-minute solution spectrum and 2-h suspension spectrum in ethanol (240-nm absorption corresponds to Ir(0) NPs).

RESULTS AND DISCUSSION

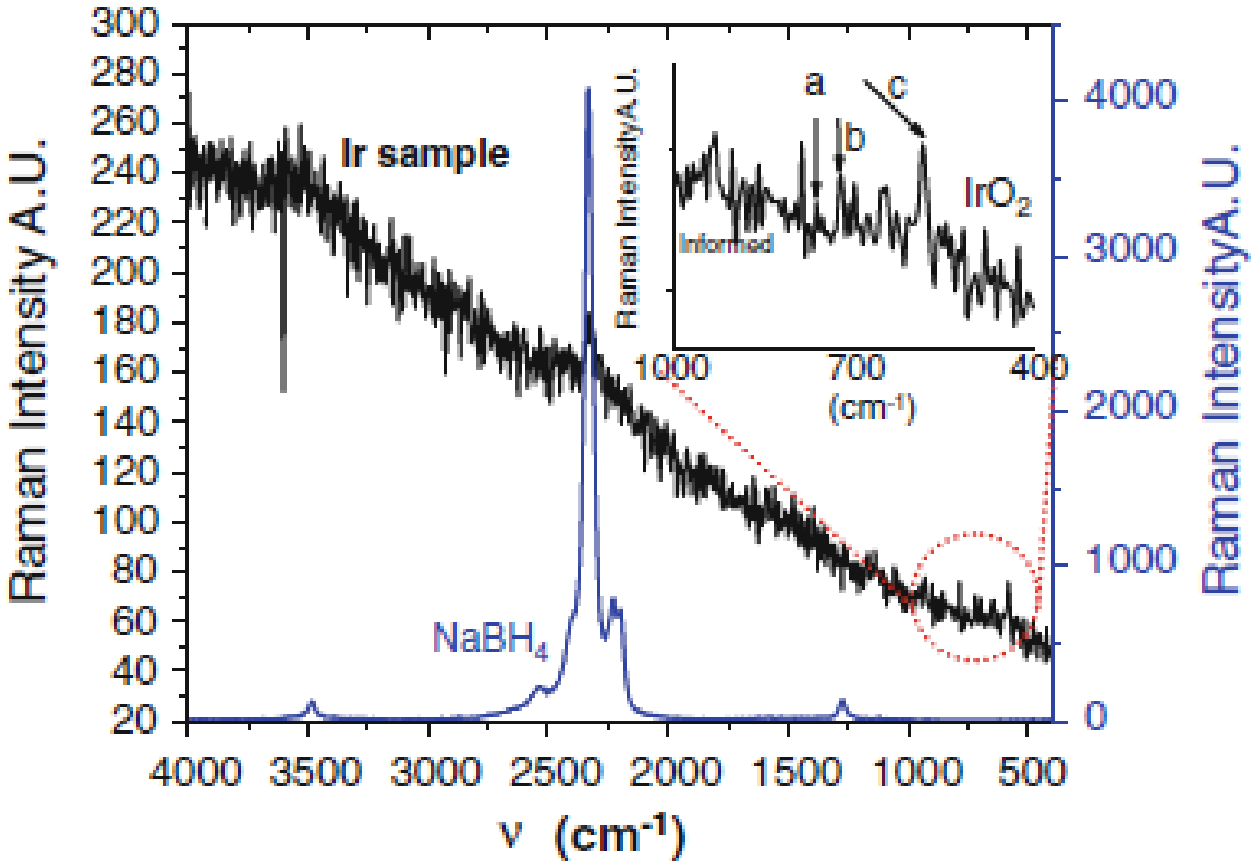

Fig. 3 Raman spectra from Ir(0) nanoparticle sample showing the contaminants; $NaBH_4$ in blue spectrum and IrO_2 in the inlet with the assignations obtained from the literature (Liao et al. 1997) a = 752 cm-1, b = 728 cm-1, and c = 561 cm-1.

RESULTS AND DISCUSSION

Table 1 Spectroscopy and size data for the Ir(0) nanoparticles obtained

$IrCl_3$ initial amount (g)	Raman[a] signals (cm^{-1})	Calc. size[b] (nm)	Stq. ratio $NaBH_4/IrCl_3$[a]	Raman[a] signals (cm^{-1})	Calc. size[b] (nm)	T[a] (°C)	Raman[a] signals (cm^{-1})	Calc. size[b] (nm)
0.05034	Weak $NaBH_4$	–	1.25	–	4.86	100	542, 714 IrO_2	8.83
0.10182	IrO_2 727,545	5.98	1.57	540,723 IrO_2	5.46	200	–	4.23
0.15215	IrO_2 542,714	8.92	2.07	540,732 IrO_2	6.80	300	540,721 IrO_2	6.94
0.20065	–	4.23	3.32	553,723 IrO_2	6.53	400	551,723 IrO_2	8.38
0.25150	–	9.44	4.20	553,723 IrO_2	20.17			
0.30223	–	18.64	5.28	IrO_2 728,561	5.13	500	545,721 IrO_2	12.6
0.35134	1602, 472–632	26.29	5.97	556,732 IrO_2	6.91			
0.40161	–	19.31	7.05	547,721 IrO_2	8.22			
			7.79	551,721 IrO_2	15.44			
			9.12	547,721 IrO_2	24.21			

Calc. size calculated particle size, *Stq. ratio* stoichiometric ratio

[a] IrO_2 Raman signals were compared with the reported (Liao et al. 1997). Raman $NaBH_4$ signals are experimental

[b] Particle size was determined using Scherrer equation (Patterson 1939)

RESULTS AND DISCUSSION

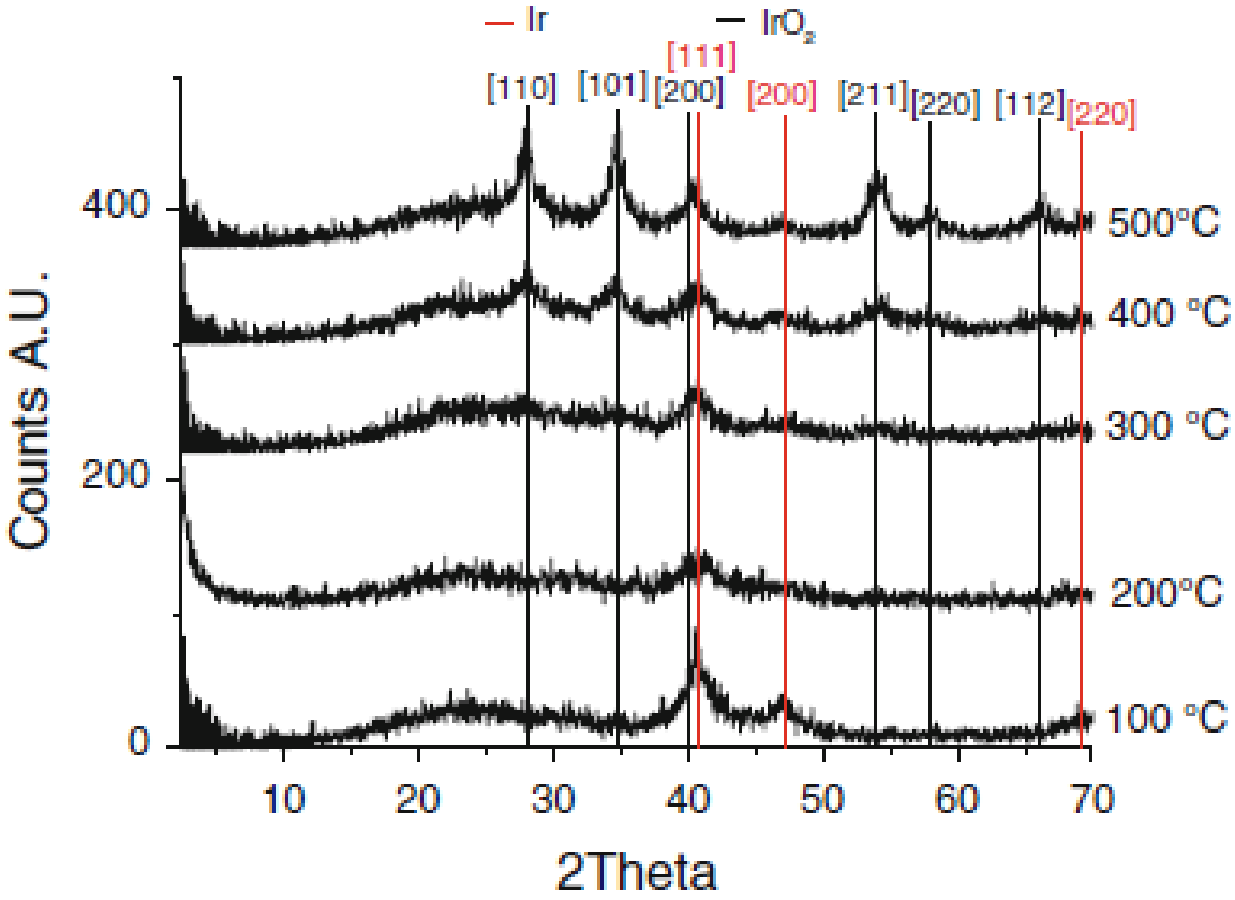

Fig. 4 X-ray powder diffraction spectra from the study of different annealing temperatures, where 200 °C is the temperature under which the smallest and cleanest Ir(0) NPs were observed. The red bars correspond to bulk Ir metal [planes (111), (200), and (220) card JCPDS-ICDD 6-598] and the black bars correspond to bulk IrO_2 [planes (110), (101), (200), (211), (220), and (112) card JCPDS-ICDD 15-0870].

RESULTS AND DISCUSSION

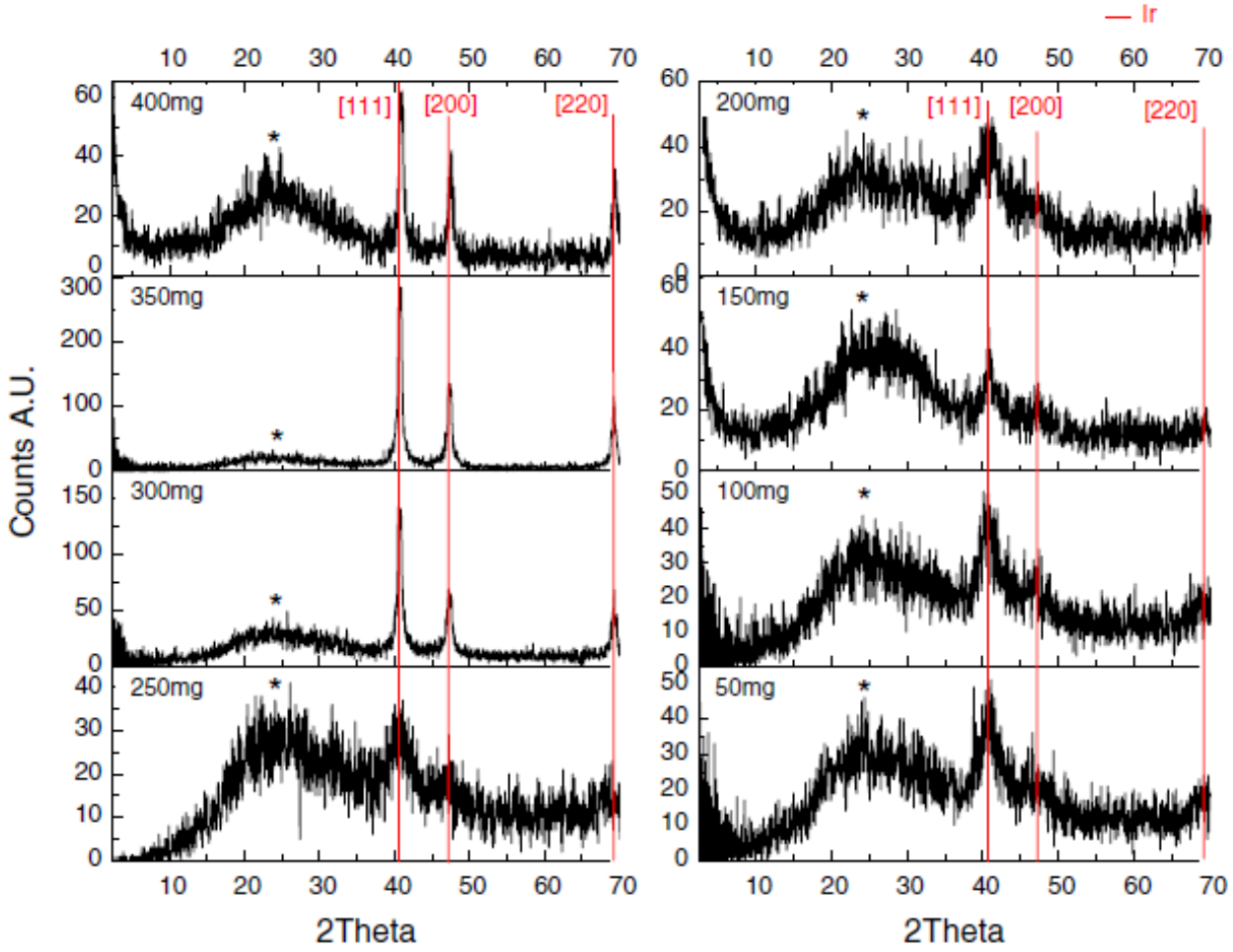

Fig. 5 X-ray powder diffraction spectra from the study of different amounts of starting Ir(III) material, where 200 mg is the amount of $IrCl_3$ with which the smallest Ir(0) NPs were obtained. The red bars correspond to bulk Ir metal [planes (111), (200), and (220) card JCPDS-ICDD 6-598]. *The bands situated around 2h = 21 and 36 correspond to the SiO_2 used as support for the X-ray diffraction experiment.

RESULTS AND DISCUSSION

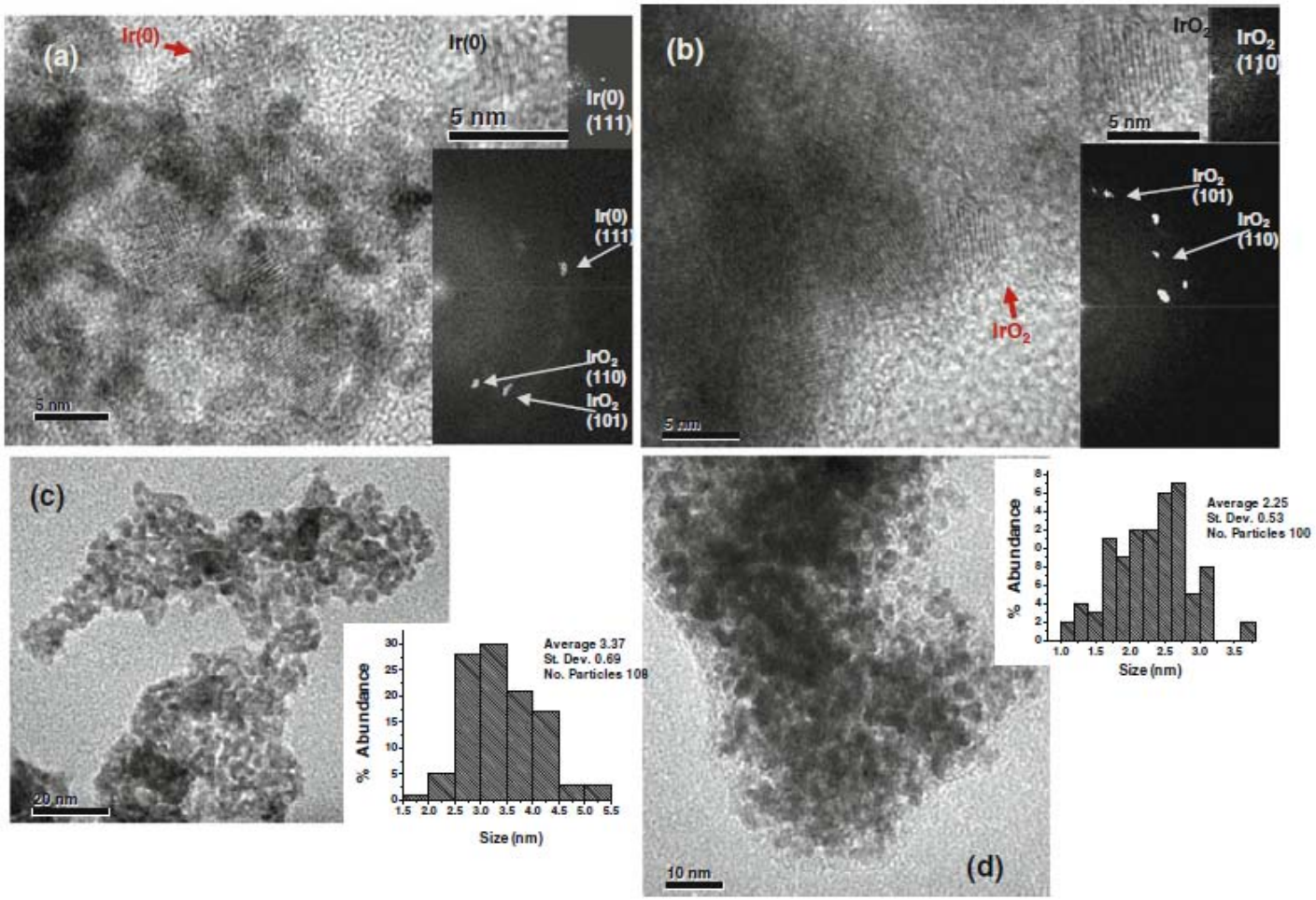

Fig. 6 a Q4 HRTEM micrography, with an isolated Ir(0) nanoparticle, its electron diffraction pattern and electron diffractogram of all particles. b Q4 HRTEM micrography, with an isolated IrO_2 nanoparticle, its electron diffraction pattern and electron diffractogram of all particles. c Q4 HRTEM micrography and its particle size distribution histogram. d Q HRTEM micrography and its particle size distribution histogram.

RESULTS AND DISCUSSION

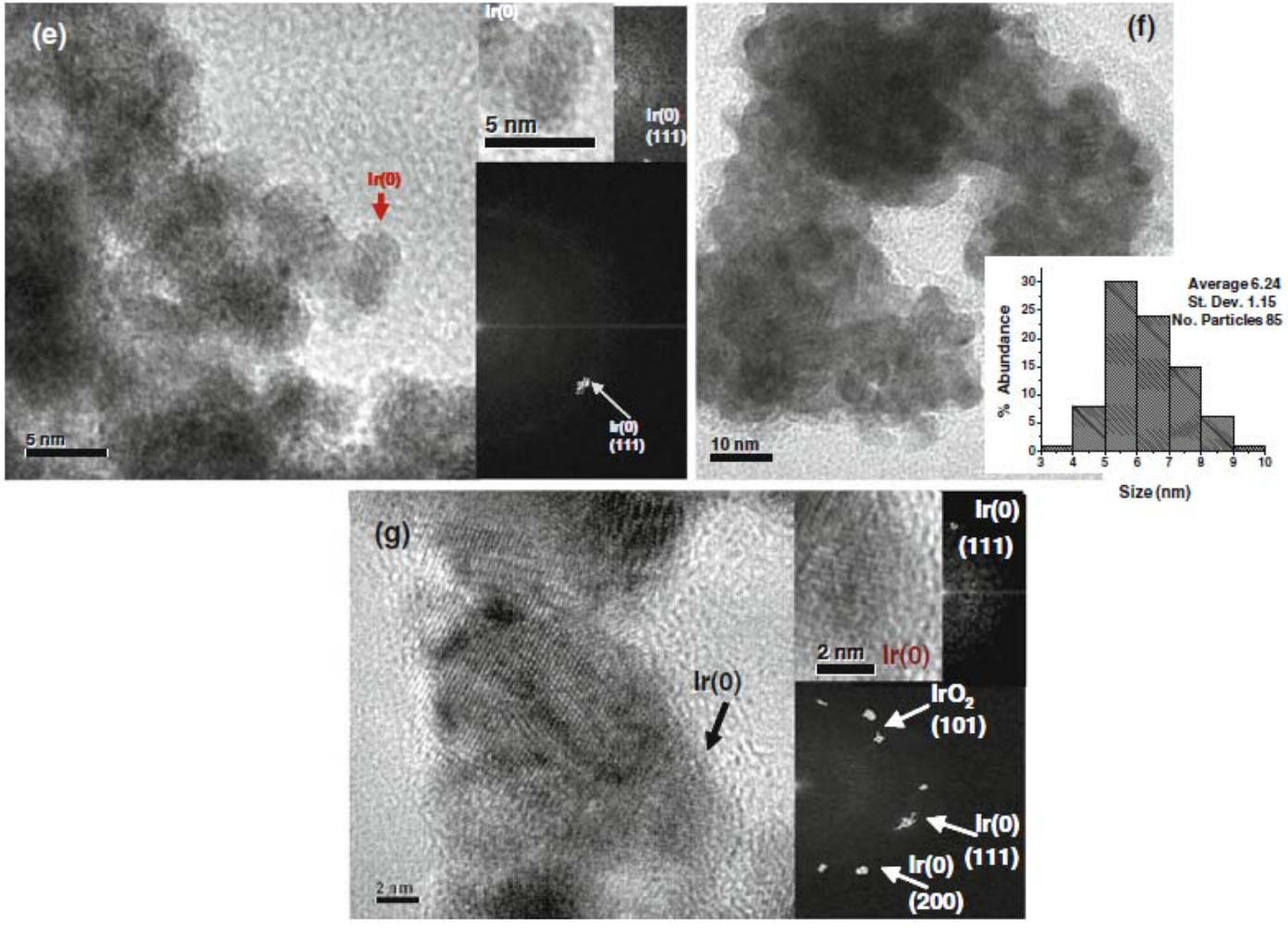

Fig. 6 e Q HRTEM micrography, with an isolated Ir(0) nanoparticle, its electron diffraction pattern and electron diffractogram of all particles. f T TEM micrography and its particle size distribution histogram. g T HRTEM micrography, with an isolated Ir(0) nanoparticle, its electrondiffraction pattern and electron diffractogram of all particles.

CONCLUSION

- An easy way to obtain Ir(0) nanoparticles under aerobic conditions was found.

- The conditions - 200 ^{o}C annealing temperature, 200 mg of $IrCl_3$ and $NaBH_4/IrCl_3$ stoichiometric ratio of 5.3 give the smallest and cleanest iridium(0) nanoparticles.

- The increase in annealing temperature increases the size of nanoparticles and also results in the production of IrO_2.

- The increase in the initial amount of $IrCl_3$ and $NaBH_4/IrCl_3$ ratio also increases the size of iridium(0) nanoparticles.

FUTURE PLAN

Synthesis of thiol protected Ir(0) nanoclusters using solid state method and their characterization.


Thank You